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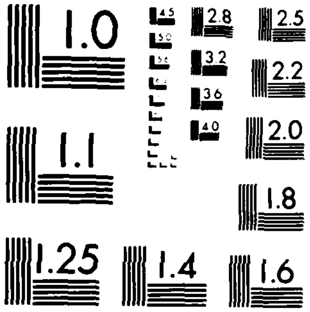

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TECHNICAL REPORT ARBRL-TR-02572

THE IMX DOUBLE DIRECT PROCESS

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August 1984

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT CENTER
BALLISTIC RESEARCH LABORATORY
ABERDEEN PROVING GROUND, MARYLAND

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The purpose of this work is to develop a new explosive based on eutectic blends of ammonium nitrate with ethylenediamine dinitrate and its homologues and to assess the safety and performance characteristics of these formulations. Various processes for making these formulations were attempted and will be described. Mixes have been scaled from several grams to 30 kilograms. Additives were introduced for stabilization, energy enhancement and sensitization. The neat eutectic is very insensitive so various additives were used to ensure detonation.

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20. ABSTRACT (continued)

A historic problem has been that of phase stabilization; a recently discovered solution to this problem, incorporation of 15 wt-% potassium nitrate with the ammonium nitrate, was used in this study. Normally, stabilizing ammonium nitrate with potassium nitrate requires working with the molten materials at elevated temperatures and processing them into prills. A unique feature of the process described in this report is that stabilization is accomplished in the melt kettle, with all other required ingredients and at much lower temperature. Formulations were evaluated from the standpoint of processing, performance, and sensitiveness. These materials appear to have great potential as military explosives because of their good performance, low cost, safety, ease of processing, and compatibility with current Army melt-cast ammunition loading plants.

The double direct process, developed here, was found to yield an explosive with less sensitiveness and less impurities than that produced by other processes.

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I. INTRODUCTION

The objective of the project on intermolecular explosives (IMX) is to demonstrate the feasibility for Army use of these materials. Associated with this effort are the tasks of selecting and developing the optimum process for production of IMX, and of testing these materials for performance and safety.

Intermolecular Explosives is a recently coined descriptor for explosives composed of separate fuel and oxidizer materials. These materials were formerly known by the term "Non-Ideal Explosives." Non-Ideal explosives were so named because their performance is not predicted by standard detonation theory. This nomenclature found little favor with the military and so was discontinued in favor of "Intermolecular Explosives." This class of explosives has many advantages. They can be made from inexpensive, abundant, available materials. They are safe in all phases of their life cycle, very insensitive, easily processible, and possess a high compatibility with current Army meltcast ammunition loading plants.

In order to introduce these materials into military inventories, many tests must be performed. These require the production of greater quantities of explosives than are normally used in the laboratory. The optimum process must be developed for the production of experimental formulations and for the production of large scale lots for military use.

The intermolecular explosives that will be the focus of this pape deal with ethylenediamine dinitrate (EDD) or its higher homologues, ammonium nitrate (AN), and necessary additives. Of principal interest will be the eutectic of EDD and AN, labeled EA, and EDD with stabilized AN, labelled EAK.

EDD was usually prepared by neutralizing ethylenediamine (ED) with concentrated HNO_3. It was manufactured by the Germans during World War II from ethanol, ammonia, and nitric acid. It was also used as cast charges in eutectic mixes with AN. Data on EDD are shown in Table I. In itself, EDD has more power and early performance than TNT, but it has less total energy, similar to nitroguanidine. In 1944, the Germans made EDD/AN eutectics (EA) for their war effort. In 1976, J. Hershokowitz and I. Akst[1] developed a solvent EA process. Scientists from Los Alamos National Laboratory and Eglin Air Force Base developed the single direct process in 1978." The Ballistic Research Laboratory (BRL) developed the double direct process in 1981. Details of these various processes will be discussed later.

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[1] *I. Akst and J. Hershkowitz, "Explosives Modification by Cosolidation of Ammonium Nitrate with Fuels," PATR 4987, Picatinny Arsenal, Dover, NJ, October 1976.*

[2] *I. B. Akst, Los Alamos National Laboratory, private communication.*

TABLE 1[3]

Ethylenediamine Dinitrate (EDD)

Mol. Wt.	186.13
Sp. Gr.	1.595 at 25/4°
M.P.	185-187°C
Sol.	Sol. in w, insol. in alc or eth
Brisance	Lower than TNT
Heat of Combustion	374.7 Kcal/mole
Heat of Explosion	127.9/159.3 Kcal/mole
Hygroscopicity at 25°C and 90% RH	1.24%
Stability	Decomposes at 270°, does not explode at 360°C

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[3] *Picatinny Arsenal Encyclopedia of Explosives and Related Items, Vol. 6, p E236, 1974.*

The inclusion of ammonium nitrate (AN) in all these systems requires some understanding of the AN, especially the volumetric changes that occur through its phase changes (Fig. 1). In the range of temperature from its melting point to below -18°C, AN has five phases,

$$\mathrm{I} \xleftrightarrow{\sim 125^{\circ}\mathrm{C}} \mathrm{II} \xleftrightarrow{\sim 84^{\circ}\mathrm{C}} \mathrm{III} \xleftrightarrow{\sim 32^{\circ}\mathrm{C}} \mathrm{IV} \xleftrightarrow{\sim -18^{\circ}\mathrm{C}} \mathrm{V}.$$

[4] The problem area revolves around the phase transition of III —— IV. This transition produces the largest volume change and creates the biggest problem for its use in both explosives and propellants. One propellant company utilizing unstabilized AN in a gas generator found that keeping the propellant extremely dry through processing and hermetically sealing the propellant in the unit prevented serious growth problems. Units stored for over 10 years in bunkers in Arizona at ambient, showed less than 1% growth and were fired successfully. Brown, et al,[5] reported mechanisms that would account for the transitions $AN_{V} \longleftrightarrow AN_{IV} \longleftrightarrow AN_{II} \longleftarrow AN_{I}$. Therefore, one method to deal with the phase III ↔ IV instability is to prevent phase III from occurring. Utilizing formulations with low moisture would inhibit the formation of phase III. The addition of moisture scavengers or the use of moisture barriers would insure safe, longterm storage.

Since moisture is difficult to exclude without incurring large manufacturing costs, better methods of stabilizing AN are required. These methods would require that phase III be locked in to provide dimensional stability over the military service temperature of -65°F to +165°F (-55°C to 74°C).

Again, in the propellant industry, potassium nitrate (KN) was found to be an excellent phase stabilizer for AN. Recent work by H. H. Cady[6] indicated that 15 wt% KN added to the AN prior to prilling is the most promising technique to maintain dimensional stability. (See Fig. 2.)

Other additives that may minimize IV ↔ III effects include NiO (4%), CuO (4%), and ternary mixtures of borates, acid phosphates, and sulfates (~0.5%).[7] All work reported in this paper with stabilized AN dealt with the AN/KN (85/15) system.

The EA (ethylenediamine dinitrate/ammonium nitrate) formulation frequently cited in this paper refers to a 50 wt-% EDD and 50 wt-% AN as the eutectic EA; the stabilized version is EAK (ethylenediamine dinitrate/ammonium nitrate/potassium nitrate). For the EAK, the weight ratios are 50%/42.5%/7.5%.

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[4] *S. D. Hendrick, E. Posnjak, and F. C. Kracak, "The Molecular Protection in the Solid State, The Variation of the Crystal Structure of AN with Temperature," JACS, Vol. 54, (1932) 2766-2786.*

[5] *R. N. Brown and A. C. McLaren, "On the Mechanisms of the Thermal Transfer in Solid Ammonium Nitrate," Proc. R. Soc London Ser A 266, (1962) 329-343.*

[6] *H. H. Cady and W. Spencer, "Ammonium Nitrate (with 15% Potassium Nitrate)/Ethylenediamine/Nitroguanidine Phase Program," Los Alamos National Laboratories, Los Alamos, NM, Informal Report ATL 9-255, 13 Feb 80.*

[7] *Report on Workshop on Ammonium Nitrate (AN) Technology, ARRADCOM, Dover, NJ, Nov 80.*

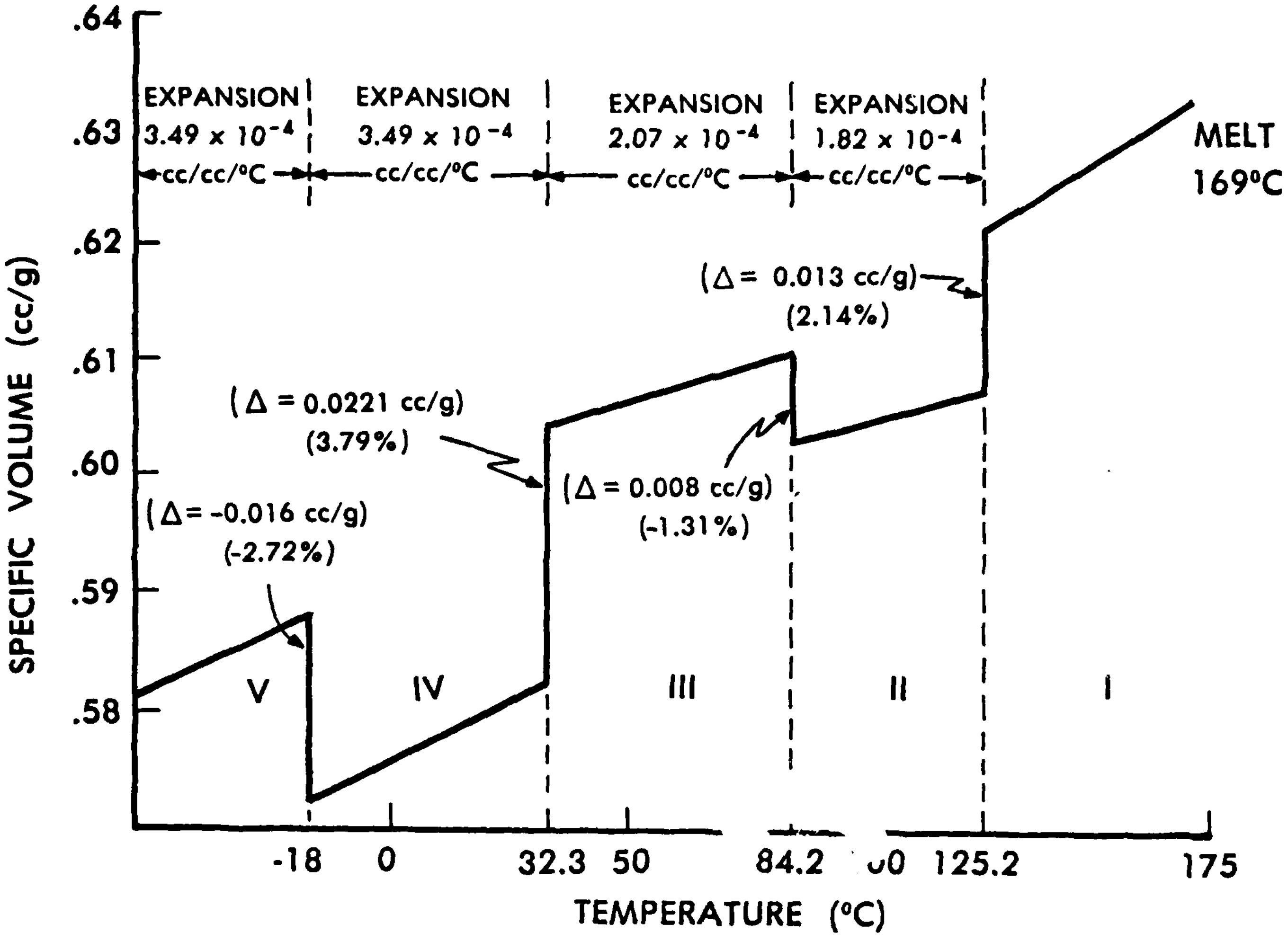

Figure 1. Phases of Ammonium Nitrate.

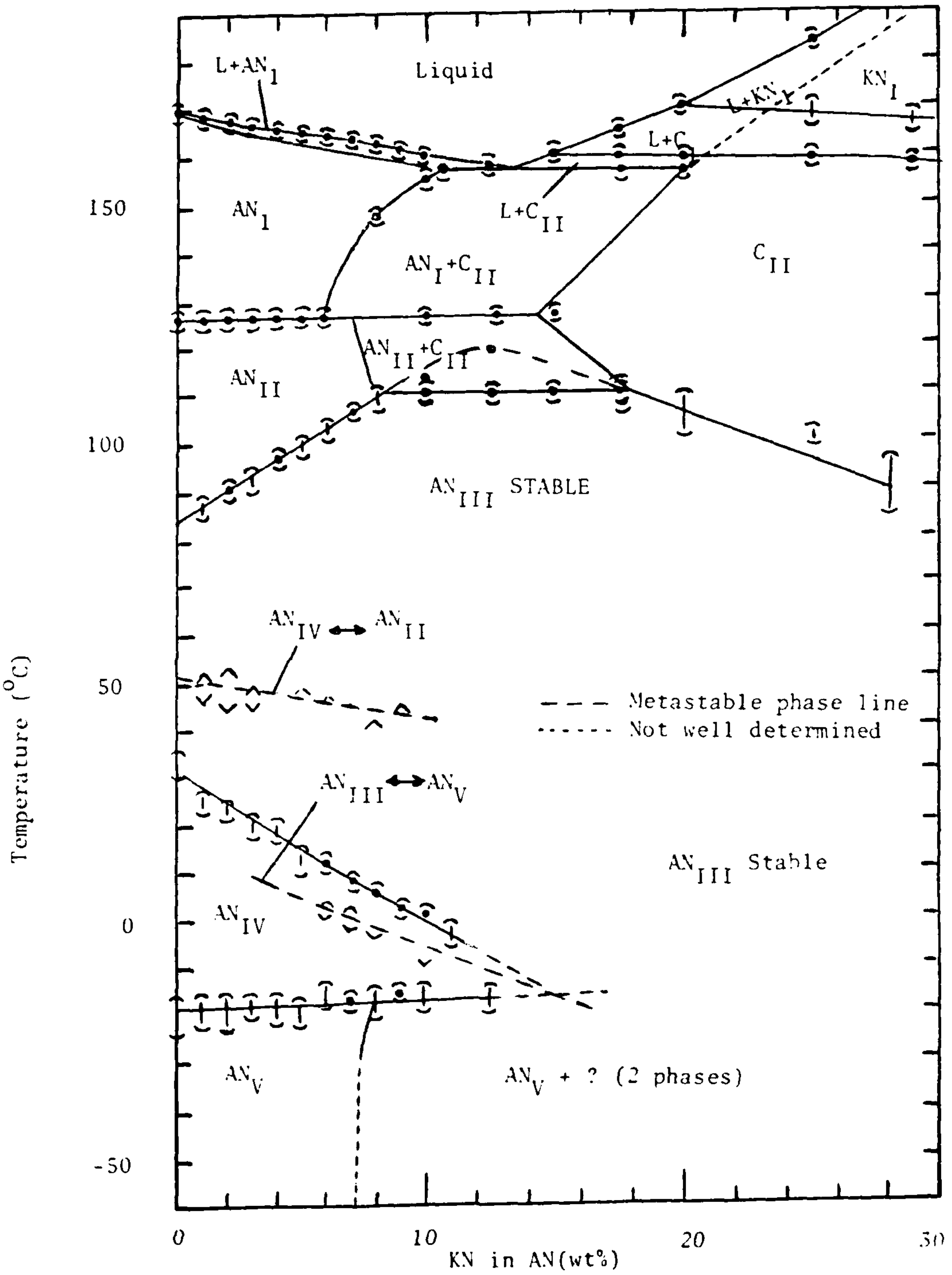

Figure 2. Phase Diagram of Potassium Nitrate in Ammonium Nitrate.[6]

This assumes that the stabilized AN with 85% AN and 15% KN acts as a single ingredient forming the binary system with EDD. Based on the reasonably close melting point data of the two eutectics, EA and EAK, this EAK can be considered as such a binary system.

Typical DSC data for IMX with stabilized AN in the composition shows no phase changes prior to melting. (See Fig. 3). H. H. Cady, of Los Alamos National Laboratory, in an internal memo[8] stated that the optimum eutectic for EA was at 32 mole % EDD and 68 mole% AN, which translates to 52.25 wt-% of EDD and 47.75 wt-% AN. The Air Force has been using approximately a 46 wt-% EDD, 46 wt-% AN, and 8 wt-% KN.

II. CANDIDATE PROCESSES

Several methods for the production of IMX's were investigated, batch sizes ranging from several grams to 30 kilograms. They are as follows: Solvent Process, Single Direct Process, and Double Direct Process. A brief description (Table 2) of these processes follows.

TABLE 2

PROCESSES FOR PREPARING IMX

A - Solvent Process

Step 1: $$\text{ED} + \text{Nitric Acid} \xrightarrow[\text{immiscible carrier fluid}]{(<60^{\circ}\text{C})\ \text{miscible or}} \text{EDD}$$

Step 2: $$\text{EDD} + \text{AN or AN/KN} \xrightarrow[\text{carrier fluid}]{\Delta\ (\sim 110^{\circ}\text{C})} \text{EA or EAK}$$

$$\text{EA or EAK} \rightarrow \text{quenched in Perclene} \rightarrow \text{filter granules} \rightarrow \text{dry}$$

B - Single Direct Process

B1. $\text{ED} + \text{AN} \longrightarrow \text{EDD} + \text{NH}_3\uparrow$

B2. $\text{EDD} + \text{AN} \longrightarrow$ Eutectic (EA)

C - Double Direct Process

$$\text{AN} + \text{HNO}_3 + \text{ED} \longrightarrow \text{EA} \longrightarrow \text{Remove Water} \rightarrow \text{Cast}$$

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[8] *H. H. Cady and W. Spencer, Los Alamos National Laboratory Internal Memo, "Ammonium Nitrate (with 15% Potassium Nitrate)/Ethylenediamine Dinitrate/ Nitroguanidine Phase Diagram."*

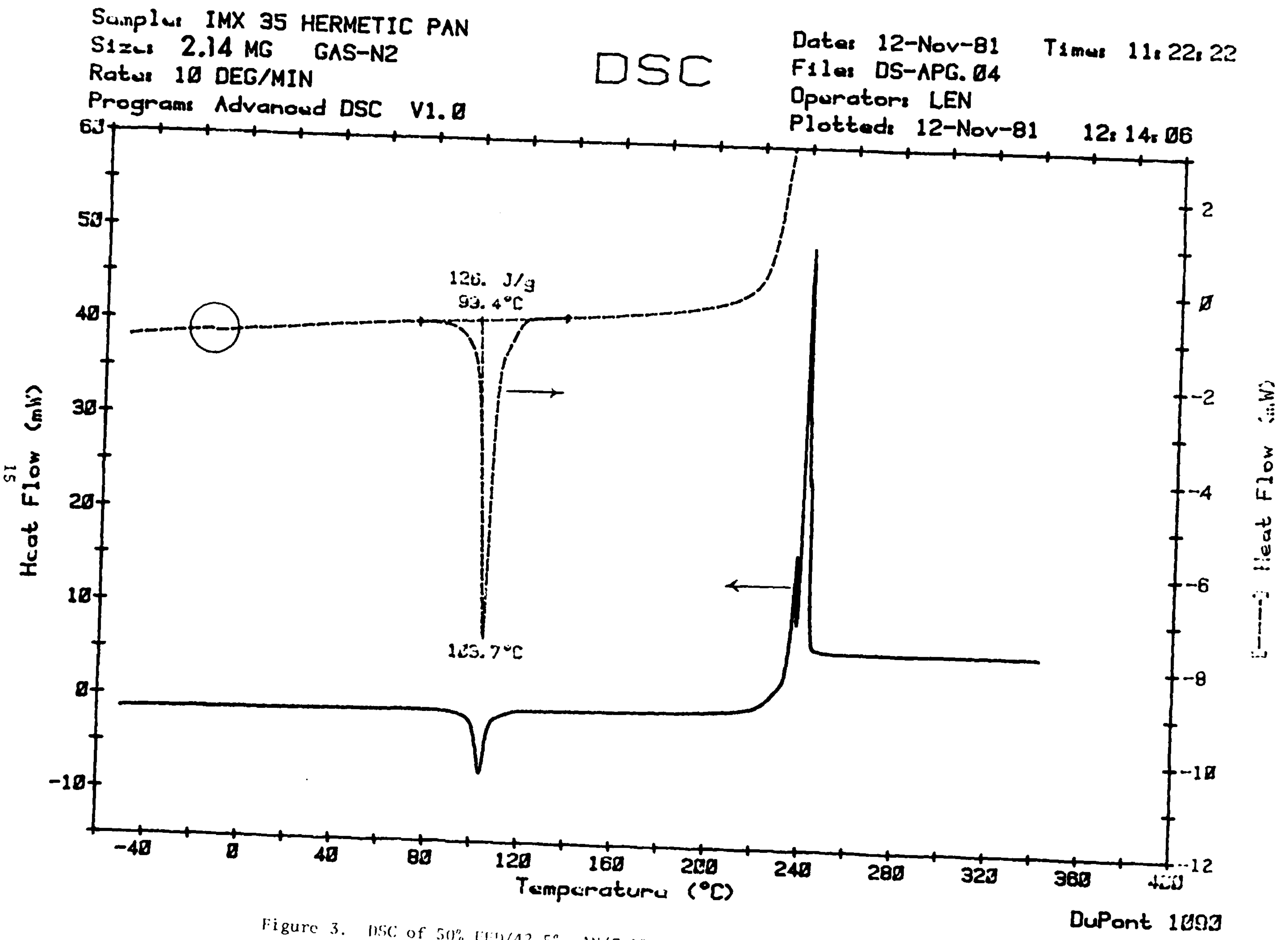

Figure 3. DSC of 50% EED/42.5% AN/7.5% KN (EAK).

SOLVENT PROCESS

Step 1 - Preparation of EDD. Perchloroethylene (PC) was placed into a jacketed reactor with the necessary quantity of ethylenediamine added to it. With the cold water running through this jacket and the stirrer going at approximately 50 RPM, add HNO_3 (70%). Requires 2 moles HNO_3/mole of ED. The reaction is exothermic. An alternate carrier fluid, e.g., alcohol, may be used instead of PC.

Step 2. Add AN or AN/KN at 1:1 ratio with EDD (reaction is endothermic) bringing up temperature to melt all solids. Pour molten EA or EAK into cold PC to precipitate granules. Filter, wash, and dry.

This process had been used by several groups to make EA. The resulting material works very well for pressed explosives, however, the process is cumbersome, requires considerable quantities of solvent, filtration, etc., and leaves residual PC and reaction products of PC and ED in the blend. These can sensitize the EA to shock initiation.[9] This can occur in the short term; it may pose a serious problem for long-term storage.

SINGLE DIRECT PROCESS

The single direct process was first done at Los Alamos National Laboratory and at Eglin Air Force Base in 1978. In this process, there is no nitric acid or solvent used. Instead, the ethylenediamine is neutralized by the NH_4NO_3 which is added incrementally. This neutralization is an exothermic reaction evolving ammonia. Two moles of AN/mole of ED are required for neutralization. The final additions of AN, when the eutectic is completely formed, should produce a 1:1 ratio with the EDD. This is an endothermic reaction, requiring heat to get it into the molten phase.

Variations of the single direct method were tried on a small scale. Method 1- Add ED to molten AN. Method 2- Add AN to heated ED. Results were similar, but temperature control was more difficult. These methods were considered more hazardous.

The single direct process is a much simpler method than the solvent process. It is also much safer because nitric acid is not used, and both starting ingredients are nonexplosive, whereas EDD, starting ingredient for step 2 of the solvent process, is an explosive by itself.

However, there is a major disadvantage to this process - 2 moles of ammonia gas are evolved for each mole of ED neutralized in this preparation, and some manages to remain in the finished product. EA has a great affinity for ammonia. Some ammonia remains even after pulling vacuum for approximately 1 hour in the molten state. Material made by this process was stored in sealed containers which were opened after various intervals of time. In all instances, the odor of ammonia and NO_x was strong. The implications for corrosivity and long-term storage are not good.

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[9] *N. Loverro, HERD Facility, Eglin AFB, FL, Internal Memo.*

DOUBLE DIRECT PROCESS

Development of the double direct process, suggested by Irving B. Akst, now at Los Alamos National Laboratory, was initiated at BRL in 1980. The process is very simple – all work can be carried out in one melt kettle. The appropriate quantities of HNO_3, AN, and KN are added to the kettle. ED is added to the kettle dropwise, stirring continuously, and controlling temperature during the addition at approximately 60°C. After the addition of all ED, the temperature is raised to 110°C; water is removed from the product by vacuum stripping. The product is then cast into desired molds.

This process has several advantages over the other processes. As in the single direct process, all starting ingredients are non-explosive and are all added to the same melt kettle. A great advantage is that ammonia is not released. The nitric acid used is at present 70% concentration -- Large Caliber Weapons Systems Laboratory, Dover, NJ, has used as high as 97% HNO_3.[10] 70% acid was selected for the sake of safety in early experiments and because the water acts as a diluent and as a heat transfer agent.

The reasons the double direct process was selected for local production of IMX are: (1) This process produces the least objectional and most easily removable contaminant -- water, which also provides a vehicle for incorporating additives and removing the heat of reaction; (2) It is easier to perform the required operations for this process than for the others -- ease of processability. (3) It has the greatest compatibility with current Army melt-cast ammunition loading plants.

Some modification of existing equipment may be required for the final water removal operation. All of these processes require 15 psig saturated steam for heating the melt kettle to exceed the 104°C melting point of EA. Most plants operate with 5 psig saturated steam, but have the capability to operate with 15 psig pressure, and current SOP's allow the higher pressure. If this problem should become significant, further research into use of the higher homologues of EDD may provide the solution. A ternary eutectic blend of DTT, EDD, and AN in the ratios 25:25:50 has its melting point around 86°C, near that for TNT and compatible with 5 psig steam processing.[2] Research is continuing on higher homologues. The use of propylene diamines with ED will also reduce the resulting eutectic melting point.

The lessons learned in working with the small-scale mixes were applied to large-scale mixes, mix numbers EAK/RD 175-11 at 15 kg and EAK/LV-1 at 30 kg. Figure 4 is a schematic representation of the arrangement of equipment for this experiment. A 10-gallon jacketed vacuum melt kettle with an anchor stirrer was used. Stirring was accomplished by an air driven motor, generally operated at about 20 psig, which resulted in a stirring rate of about 50 RPM. No vacuum was applied during addition of ingredients and while reactions were occurring. After all reactions had taken place, the temperature was raised and vacuum applied to remove the water. The procedure used for making a 30 kg mix of EAK by the double direct method is detailed in Appendix A.

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[10] *W. Voreck, Large Caliber Weapon Systems Laboratory, Dover, NJ, SOP EMD-245, Feb 81.*

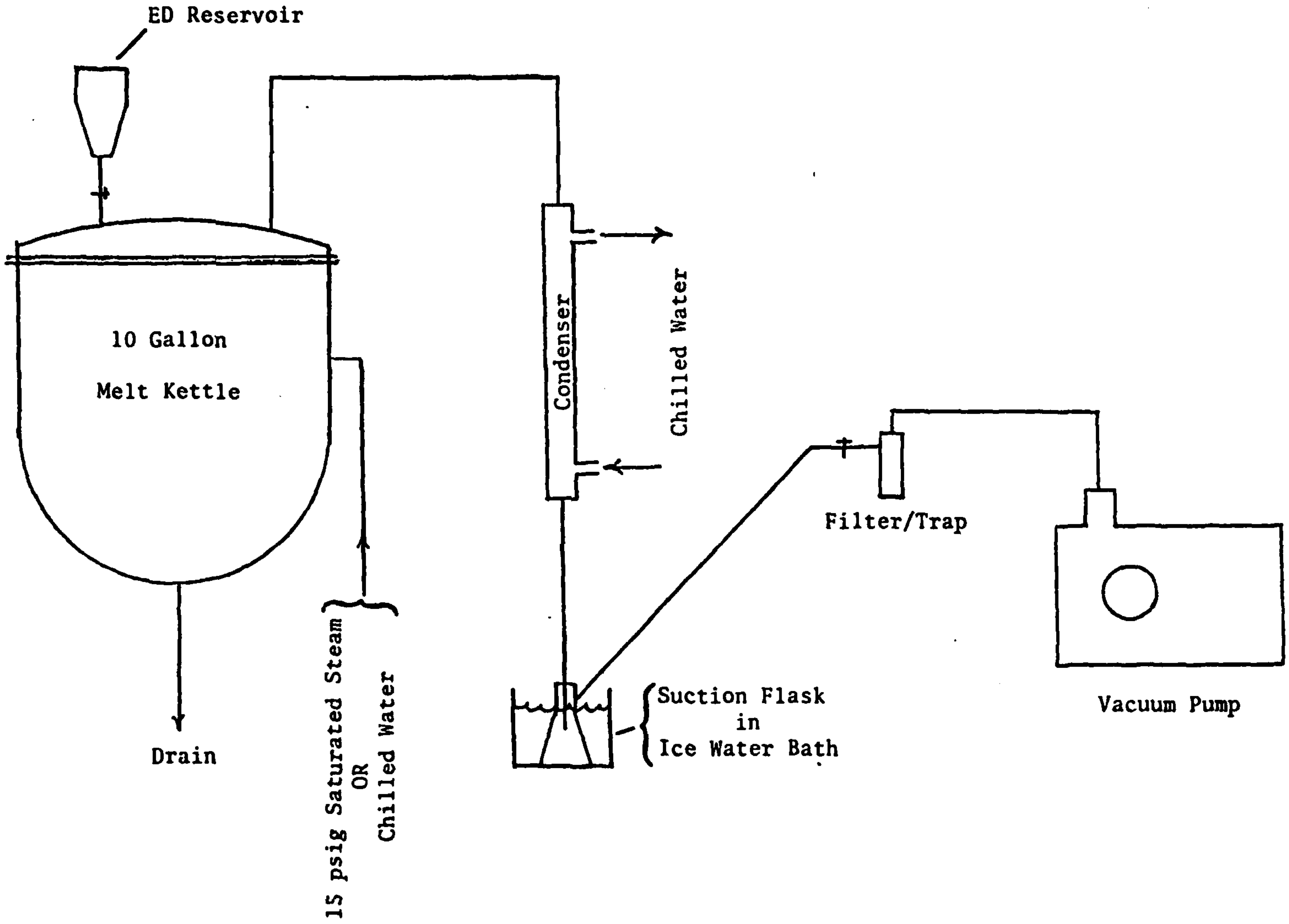

Figure 4. Experimental Configuration for 30 kg Mix.

III. ADDITIVES

To improve the explosive properties of the IMX compositions, it was necessary to make them slightly more sensitive. This could be achieved several ways. One method would be to introduce nucleating sites via such materials as extremely fine Al_2O_3(preferably the fumed alumina with surface areas ~100 square meters per gram),glass microspheres or colloidal silica. The latter was chosen, and M-5 Cab-O-Sil was added to EAK. It was found that 4 wt-% Cab-O-Sil is the maximum in an IMX composition that will permit routine casting. Higher percentages thicken the mix excessively. Formulations with 3.5% and 3.8% will be discussed in the test section. Densities of IMX compositions containing Cab-O-Sil were over 95% of TMD.

Another method of improving the explosive properties of IMX is to enrich the formulation with energetic materials such as RDX, HMX, etc. RDX was selected for test purposes. Some difficulty was encountered using dry Class 5 RDX as the enriching agent at the 25% level. These compositions had densities as low as 67.7% of TMD. A novel method was developed that improved the processing considerably. A small four-bladed impeller was substituted for the conventional stirrer used in hot melt operations. The stirring speed was increased from 50 to 250 RPM. This was done to incorporate the RDX intimately into the IMX. However, the higher speed stirring whipped the composition into a foam. It was found that 3M's Fluorad FC 170-C wetting agent at 0.1% broke down the foam quickly and allowed the processing of the formulation to proceed normally. Final density was better than 97% TMD.

Adding the combination of Cab-O-Sil and RDX provides another method of improving IMX detonation properties with minimal processing changes. Before the water is removed at the end of the double direct process, 2% Cab-O-Sil and the desired quantity of RDX are added directly to the kettle. The Cab-O-Sil serves a dual purpose-- On one hand it sensitizes the composition; on the other hand it acts as a thickening agent, allowing the suspension of a wide range of particle sizes of energetic materials, such as RDX. These may be added in the as-received state, i.e., moist. There are other methods of incorporating solid energetics, usually as dry powder, into an explosive where particle density and size become factors. These methods are called "creaming"; they rely heavily on precise temperature control to effect suspension. Creaming will be discussed later.

The safety in handling RDX during processing was also improved. It was found that if the EA or EAK is made first and then RDX added as received(wet) good product (densities better than 95% TMD) was made. For this method, the wetting agent is necessary.

Other means of improving IMX may be the inclusion of minor quantities of transition metal compounds that form sensitized complexes in situ. Wetting agents such as Fluorad 99 and DuPont Zonyl FSK were very effective in dispersing Cab-O-Sil and RDX in IMX. Dow Corning DB 100 was used to minimize frothing during the vacuum stripping phase of the process.

A combination of both sensitization and enrichment was accomplished without the aid of the wetting agent. A density of 96.5% TMD was realized from a mix of 2% Cab-O-Sil and 1.5% RDX (Class 5). Details will be reported in the test section.

DISPERSING AGENT/WETTING AGENT STUDY

A series of tests was run to determine the best wetting agent for incorporating wet, as shipped, RDX into EAK. Some of these agents require the presence of water in the EAK, others are more effective without water. Table 3 lists the wetting agents with indications of how well the RDX disperses in the EAK and how well the tendency of the EAK to foam during vacuum distillation is controlled.

Table 3

Wetting Agent Performance for RDX in EAK[a]

Wetting Agent	Manufacturer	Dispersion	Defoaming
FC-170-C	3M	3	5
FC-171	3M	4	6
Zonyl FSK	DuPont	2	4
Zonyl FSN	DuPont	5	3
Zonyl FSC	DuPont	6	2
FC-99	3M	1	1

[a] Rankings are by visual observation. No. 1 is best.

Scanning Electron Micrographs (SEM's) were made for each surfactant tested. Samples were prepared for SEM observation by cutting and polishing them. Successively finer grades of abrasive paper were used, wet with silicone oil. This was followed by polishing with polishing cloths, first wet with silicone oil, then wet with water. The final polish was done with water to dissolve out some of the salt and leave the RDX in relief. Figure 5 shows a set of three SEM's at different magnifications of one of the samples, IMX with DuPont Zonyl FSN surfactant. 50 g of IMX were used: 48.25 g EAK/1 g Cab-O-Sil/0.75 g RDX, together with 7 ml of 0.1% Zonyl in water. The RDX appears to be well dispersed throughout the EAK, and very little clumping can be observed.

The observations described in Table 3 were made in connection with the preparation of Plate Dent Test (PDT) specimens. These specimens were fired to determine the effect of these surfactants on performance. The results of these test firings will be discussed with the other plate dent test data.

CREAMING TESTS

Normally additives e.g., Cab-O-Sil, have been used to suspend solids in the EAK explosive. There are processing methods which can be very effective also in suspending solids in liquids; creaming is one of these, and this is treated briefly here. Air Force personnel at the HERD Facility, Eglin Air Force Base, Florida, have used a creaming method to suspend H5 aluminum spheres in EAK.[11] This method involves melting the EAK, mixing in the aluminum spheres, and lowering the temperature to just above the freezing point. Three cycles of freezing and melting are then performed in rapid succession. During the freezing phase, a crust is formed on the inside surface of the kettle, and during the melting phase this crust is sloughed off and re-incorporated into the melt. Three cycles of this yield a slush having a higher viscosity than the EAK had prior to this procedure and having the capability of suspending particulate matter.

The Army, at the Ballistic Research Laboratory (BRL), has long been creaming TNT for grain size control and for aesthetic reasons. In this process, TNT is melted and brought to a temperature just above the freezing point. Powdered TNT is then stirred into the melt and the product is cast. Again, this yields a slush with an increased viscosity. Color and texture of the product were improved by this procedure.

Both of these procedures were performed at the BRL on EAK made by the double direct process, without particulate additives. The Army method required only 3 wt-% of powder to produce the desired consistency of EAK. Both methods yielded good quality castings; the method of choice will depend upon available facilities and operator preference. Further tests are planned; it is reasonable to expect to be able to suspend class 1 RDX in creamed EAK without the need for suspension agents like Cab-O-Sil.

PLASTICIZING AND PROCESSING TEMPERATURE REDUCTION

In the round-to-round communication tests, which will be described later, it became apparent that neat EAK was more sensitive to impact than was desired. This is likely because this material is very hard and therefore subject to shear initiation. Several additives are being considered to plasticize EAK, to soften it, and thus reduce its sensitiveness to shear. One of these is urea nitrate, formed by nitrating urea with nitric acid. The nitrate salt melts with decomposition over a broad range, 135 to 165°C. It has a strong exotherm at 167°C.

Urea nitrate was melted with EAK. It was necessary to heat the mixes to 140 or 150°C in order to blend the urea nitrate into the EAK. Mixes containing 17, 40, and 50 wt-% urea nitrate were prepared. The 17 and 40% urea nitrate mixes were prepared at 140°C and had melting endotherms at 89°C and 65°C, respectively. The 50% urea nitrate mix was prepared at 150°C and was a waxy solid with a broad melting range from room temperature to 120°C. A larger scale preparation made with 50% urea nitrate was a somewhat soft solid, like fudge.

Performance of EAK/Urea Nitrate formulations has not been assessed, however replacing a portion of EAK by urea nitrate will likely not adversely affect its performance because urea nitrate is nearly oxygen balanced.

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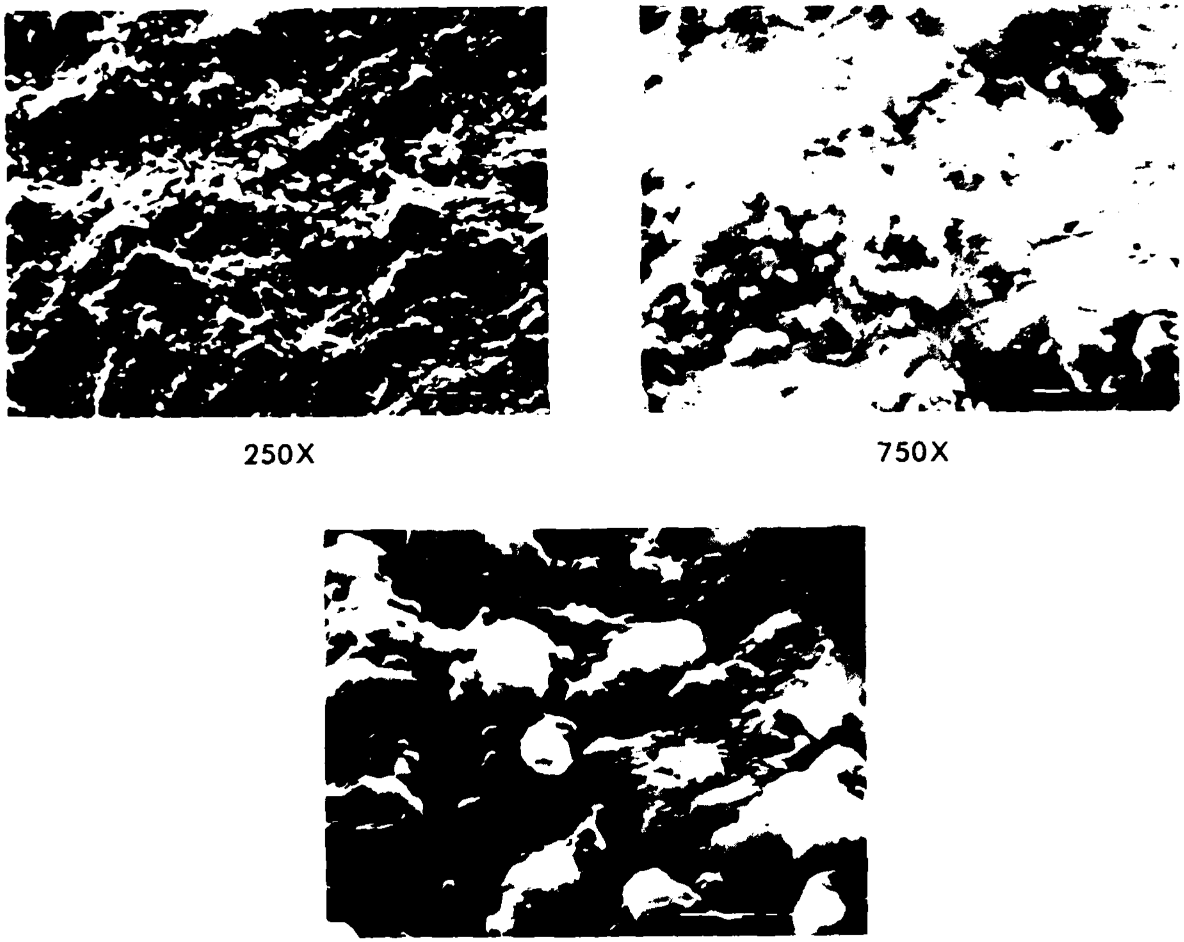
[11] *G. Parsons, HERD Facility, Eglin AFB, FL, private communication.*



250X

750X

1500X

Figure 5. SEM's of EARK-1.5 with 2% Cab-O-Sil, Showing RDX Dispersion.

IV. TEST RESULTS

The tests that are described in this section all used the locally produced IMX, with a density of 1.610, 96.7% TMD, and about 0.5% water, made by the double direct process described earlier. The formulation used was EDD/AN/KN (EAK) in the ratio of 50/42.5/7.5 by weight. Figure 3 shows a DSC run on this material in a sealed pan. The sealed pan prevented the endotherm which would be caused by sample evaporation. The reason for including the 7.5 wt-% KNO_3 was to phase stabilize this blend. The DSC of Figure 3 indicates that this was completely successful. There is a perturbation in the expanded trace (upper curve) ca - 16°C. Even if it has physical meaning, it is too small to be significant. Also, the endotherm at the eutectic point occurs just where it ought, at 103.7°C. The discontinuity in the exotherm occurred because the sample pan ruptured. The onset of the decomposition exotherm occurred beyond 230°C.

Early in the effort, three types of performance test had been performed: Prompt Energy Test (PET), Plate Dent Test (PDT) and firings of larger, bare charges, Large-Scale Tests (LST). None of these tests was very sophisticated, but they did provide much useful information for screening various formulations with respect to their performance. Since then, many other tests have been performed to assess both safety and performance. Tests performed thus far are listed in Table 4.

TABLE 4

Performance and Safety Tests on IMX

Performance	Safety
Prompt Energy	Drop Weight Impact
Plate Dent	Friction
Large Scale-Bare Charge	Fratricide
Cylinder Expansion	Activator
Fragment Speed	
[a] Sandwich	

[a] This is a non-standard test, performed for a particular application.

These tests and their results are discussed below.

PROMPT ENERGY TESTS

One of the performance parameters of an explosive is the rate of energy release early in the detonation, the prompt energy. A measure of this is the degree of fragmentation of the containment vessel that results from a detonation. Steel cylinders, 6" long, 2-1/2" I.D., 3" O.D., were filled with TNT, and with composition-B, and fired as calibration shots. Neat EAK was tested in the same manner. Data were generated as histograms of the number of fragments per mass increment versus mass. Figure 6 shows the calibration histograms, and Figure 7 that for neat EAK. Composition-B produced significantly smaller fragments than TNT, the crossover occurring ca. 2 g. The neat EAK showed far fewer small fragments than either TNT or composition-B. This EAK charge likely didn't detonate.

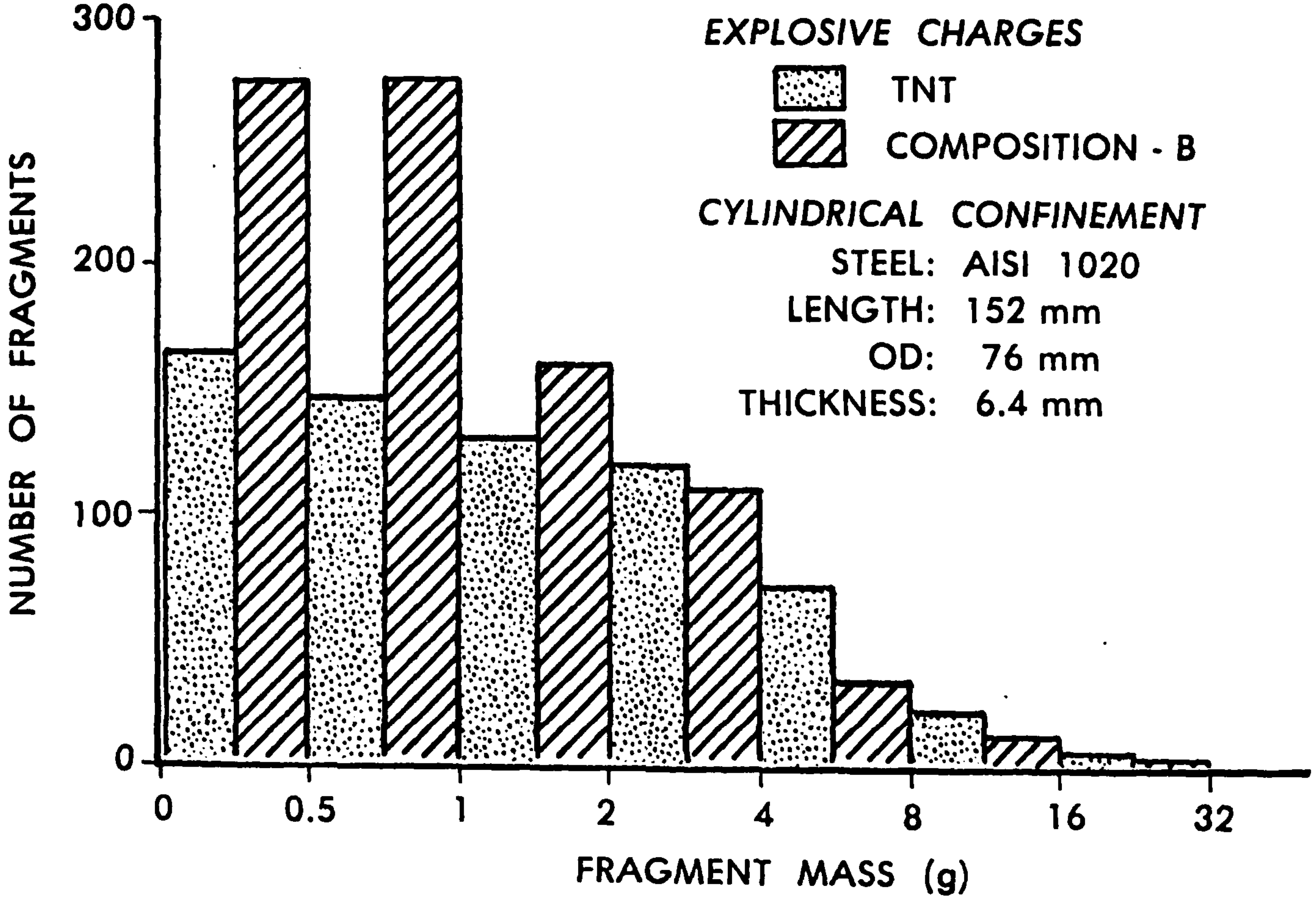


Figure 6. Calibration Tests for Prompt Energy Tests.

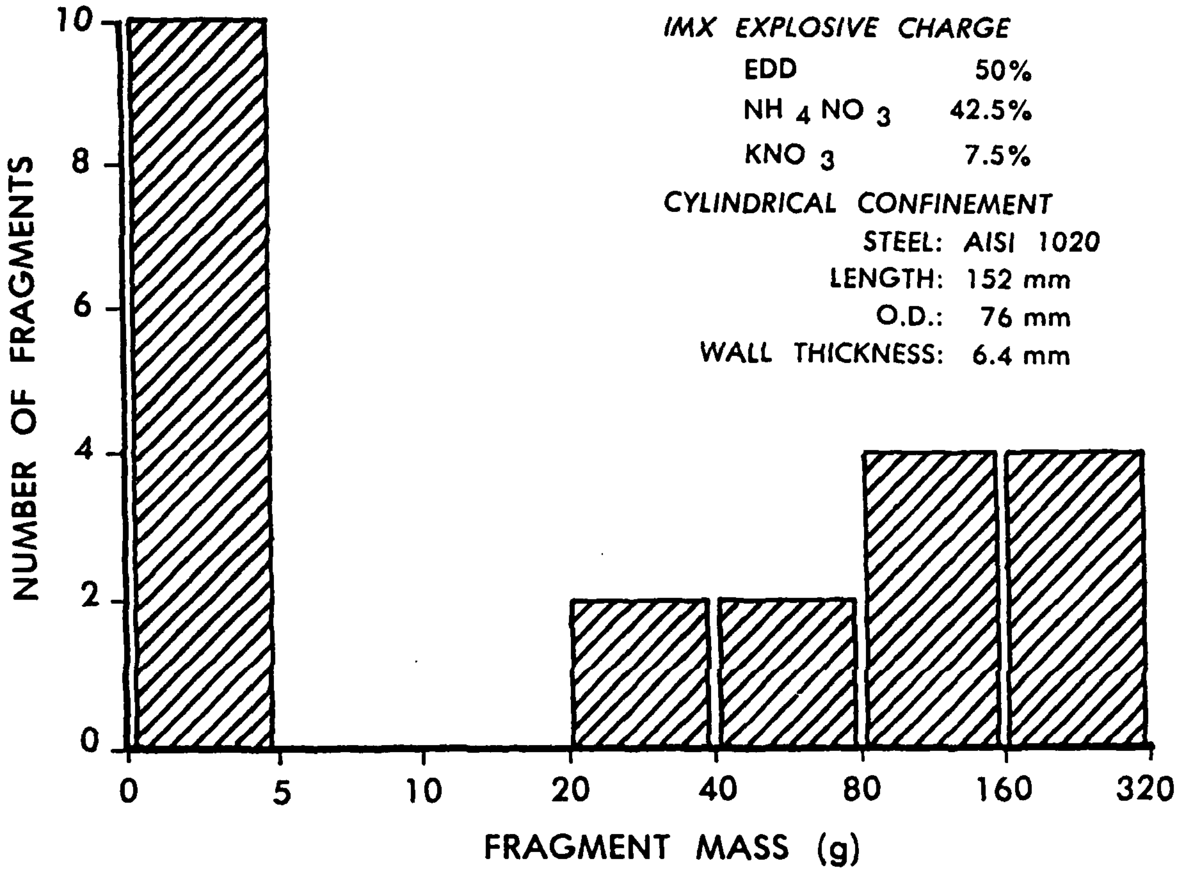


Figure 7. Fragment Histogram for Neat EAK Showing Low Brisance.

PLATE DENT TESTS

The plate dent test is another performance screening test. The experimental configuration used initially is shown in Figure 8, and a summary of results in Figure 9. An earlier PDT used neat EAK as for the PET described above. Again, there was no detonation; a very slight dent was observed in the witness plate. This test did point out the very insignificant contribution to the witness plate deformation from the booster and remainder of the firing train. The base relief cylinder was used to emphasize the effect of the main charge. That it was unnecessary is demonstrated in Figure 9 – holes were formed in the witness plate whenever detonation occurred. Since neat EAK produced at the BRL had not yet been detonated, 25% RDX was added as an enriching and sensitizing agent. Class 1 RDX was initially used, but settled out of the molten EAK too rapidly; thus,class 5 RDX was used. This proved difficult to blend with EAK. The RDX used for shot #1, Figure 9, was hand incorporated into the EAK. Vacuum oven outgassing was not used. As a result, the charge which should have weighed 6 oz weighed only 4 oz, yielding a density of 67.7% TMD. As can be seen, it detonated very well. A plug of steel from the witness plate was driven into the base relief cylinder.

Shot #2 represented an attempt to reduce the amount of RDX used, and yet achieve detonation through sensitization of the EAK without significant enrichment. 1.5% class 5 RDX was used, along with 2% Cab-O-Sil to hold it in suspension during freezing. A considerably better density was obtained, 95.9% TMD. The PDT performance was about equal to that of shot #1, however, 50% more HE was used because of the greater density.

Shot #3 was done, using composition-B, for comparison purposes. Performance for this shot was better than for shots #1 and #2. However, in comparing shots #1 and #3, it appears that shot #3 is a reasonable extrapolation of shot #1, given a 50% increase in HE; shot #3 had 50% more HE than shot #1.

Another formulation was tested similarly: 96.5% EAK/3.5% Cab-O-Sil. The intent was to ascertain whether Cab-O-Sil, a non-energetic thickening agent, could by itself sensitize the EAK. Detonation did occur; this detonation punched a hole in the witness plate, but this event did not appear to be as vigorous as the previous shots.

It became obvious from these tests that the experimental configuration would require modification in order that the data could provide a means of assessing performance of IMX formulations. The new design is similar to the previous design: The mild steel witness plate thickness was increased from 1" to 3", and the base relief cylinder was removed. Subsequent tests were fired with the confined main charge resting on the 3" thick steel witness block, which in turn rested on the steel floor of the firing chamber. Table 5 lists the results of these tests. Calibration tests were performed first, with TNT, Comp-B, and Octol. These three explosives were thought to encompass the performance range to be expected from IMX's. Three shots of each were fired, and a significant range of dent performance was observed for the three types of explosives. The next series of shots used RDX-enriched EAK in an attempt to achieve Comp-B equivalent performance. The best dent recorded was 8.95 mm, just smaller than the 9.2 mm Comp-B dent. Further improvement appears possible, apparently without using greater proportions of RDX. Refinements in processing will likely yield the desired performance.

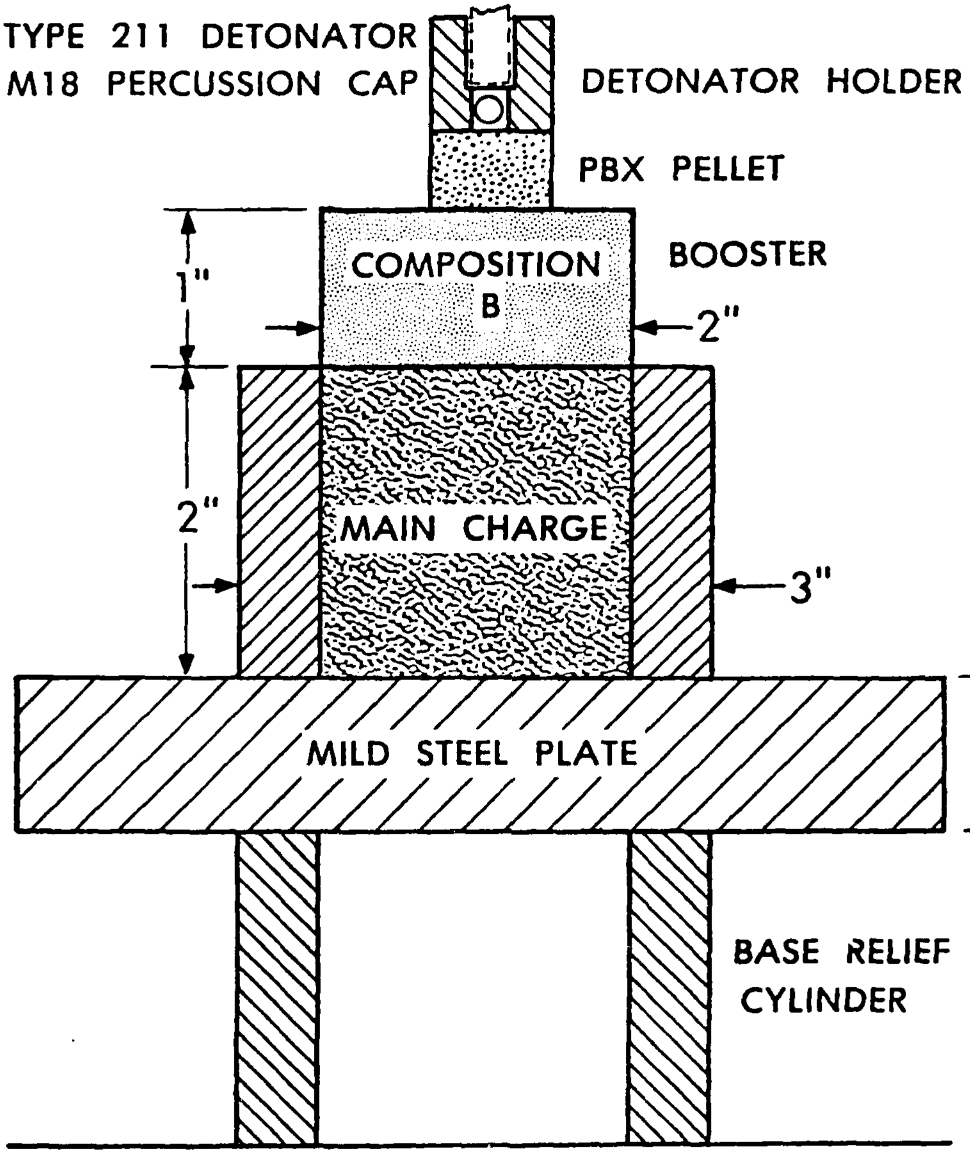
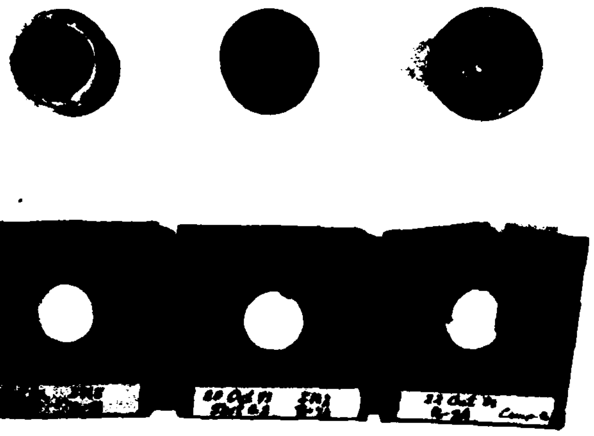


Figure 8. Experimental Configuration for Plate Dent Tests.



FOR ALL SHOTS: IMX ≡ EAK (50/42.5/7.5)

SHOT # 1: 75% IMX/25% RDX (CLASS 5)
ρ = 67.7% TMD (~4oz. CHARGE)

SHOT # 2: 96.5% IMX/2% CAB-O-SIL/1.5% RDX
ρ = 95.9% TMD (~5.9oz. CHARGE)

SHOT # 3: COMPOSITION-B (FOR COMPARISON PURPOSES)

Figure 9. Plate Dent Test Results - IMX Performance Evaluation.

TABLE 5

PLATE DENT TEST RESULTS
(NO BASE RELIEF   3" THICK WITNESS PLATES)

EXPLOSIVE	CONCENTRATION	DENSITY	%TMD	DENT
TNT	(RDX/TNT) 0/100			7.6
COMP-B	60/40			9.2
OCTOL	75/25			10.4
IMX	(RDX/EAK) 21.9/78.1	1.279	75.5	5.70
"	"	1.243	73.4	6.01
"	"	1.237	73.0	6.17
"	"	1.517	89.6	8.54
"	"	1.568	92.6	8.95
"	"	1.578	93.2	8.87
"	25/75	1.670	98.3	8.45
"	20.7/79.3	1.690	99.9	8.85
"	20.4/79.6	1.539	91.0	8.85
"	24.1/75.9	1.641	96.7	8.37
"	24.6/75.4	1.606	94.6	8.13
"	21.2/78.8	1.545	91.3	7.78
"	(EAK/CGS/RDX)96.5/2.0/1.5	1.577	94.1	6.88
"	"	1.601	95.6	7.19
"	"	1.590	94.9	5.80

TABLE 5. Plate Dent Test Results.

The last three shots tabulated here were performed in order to demonstrate that sensitized EAK could perform as well as TNT. The 7.19 mm dent approaches the 7.6 mm TNT dent but still falls too far short of it. Sensitizers other than Cab-O-Sil (COS) will be tested, and the proportions used in the formulation will be varied. These changes, together with processing improvements are expected to produce the desired results.

LARGE-SCALE TESTS

Two tests were run, in which 5 inch diameter neat EAK charges were made and fired. one with a length of 5.5 inches, the other 11.2 inches. The smaller of these was poorly instrumented; the principal effect observed was the cratering of a lead block by a single, prepared, $(1\text{-cm})^3$ steel fragment, imbedded at the opposite end of the charge from the detonator, in a 1-cm thick disc of Wood's Metal covering the entire end of the charge. This charge was initiated, it reacted violently but did not fully detonate. The Wood's Metal did not vaporize as expected, but created many peripheral craters in the lead block. The central crater, that was produced by the prepared fragment, had a volume of ca. 6 cc. This scaled to an impact velocity of 300-500 m/sec; $\sim$ 1800 m/sec had been expected if full-scale detonation had occurred.

Because it was thought that a 5 inch diameter bare charge of neat EAK should be above the critical diameter for detonation, another charge was prepared, having the same diameter, but an increased length, 11.2 inches. It was more completely instrumented than the first such test (see Figure 10). The charge was mounted vertically in a test barricade. The base of the charge was configured as before, a steel fragment in a Wood's Metal surround, aimed at a lead block. X-ray tubes were arranged to measure fragment speed. Detonation velocity was determined by sensing with piezoelectric pins whose signals were summed and recorded on a Biomation Waveform Recorder. Figure 11 shows the results and the summing network used. Detonation was initiated by a very large booster, 5 inch diameter x 3 inch long composition-B. When the detonation wave reached the first pin, it had slowed considerably, but was likely still chemically driven because of its speed and the fact that no unreacted EAK was found. Pins 1, 2, and 3 were located 9 cm, 5 cm, and 1 cm, respectively, from the base of the charge. The speed of the detonation wave was 3.37 mm/μsec between pins 1 and 2, and 3.07 mm/μsec between pins 2 and 3. Since detonation velocities of ca 8 mm/μsec have been observed by others,[2] we can conclude that even this large charge did not fully detonate. Further tests were not made to determine the critical diameter of neat EAK produced by the double direct process. For all but the largest charges, this explosive will need sensitization. However, the enrichment required to yield adequate performance for many applications also provides the required sensitization. For low performance HE, small quantities of impurity (e.g., Cab-O-Sil) and RDX may suffice, as indicated by the plate dent tests.

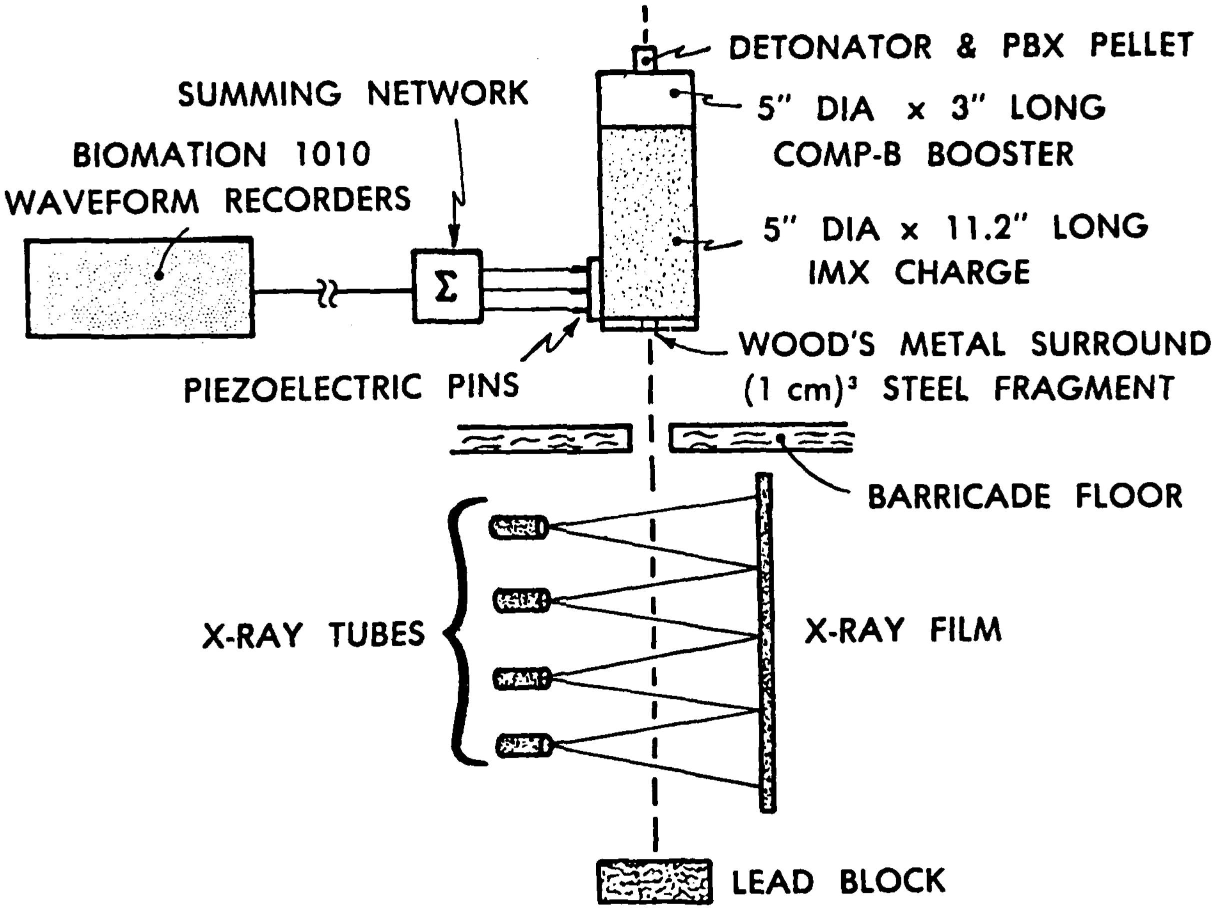


Figure 10. Experimental Configuration for Large-Scale Test of Neat EAK.

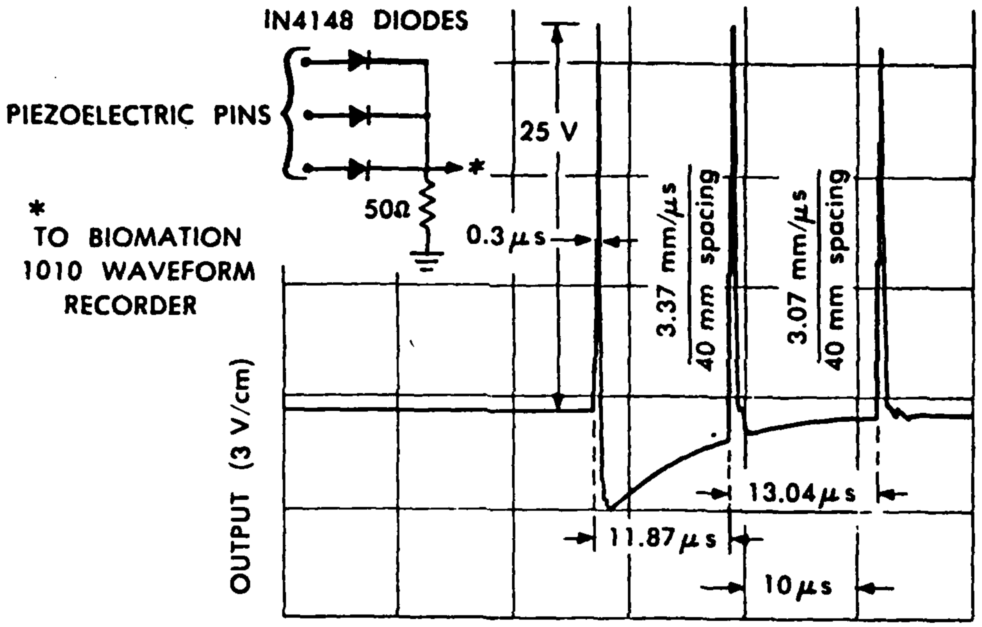


Figure 11. Results of Large-Scale Test Firing of Neat EAK.

ACTIVATOR TESTS

The activator test was designed to determine sensitiveness of explosives to hot spot initiation such as could occur during projectile launch. The explosive fill in a shell experiences inertial setback forces upon gun launch which can act upon voids in the explosive. Adiabatic compression of the gas in these voids causes intense, local energy deposition; i.e., small regions of high temperature; these hot spots can initiate chemical reaction in the explosive which can grow to detonation.

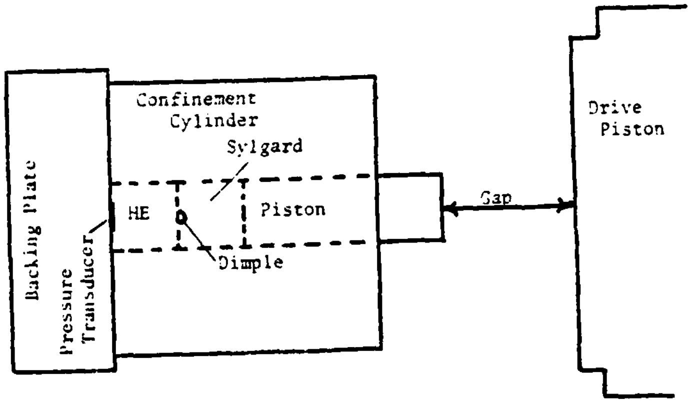


Figure 12. Activator Test for Setback Sensitiveness Measurements.

These tests are performed as follows (see Fig. 12): 1/2 inch diameter by 1/2 inch long cylindrical samples are prepared. One of these is inserted into a heavy steel confinement cylinder, with a pressure transducer between its base and a heavy steel backing plate. A 1/2 inch long Sylgard cylinder with a small dimple in it base is seated against the explosive. A 1/2 inch diameter steel piston is then seated against the Sylgard, with its far end extending well beyond the end of the confinement cylinder. A short (variable) distance away from this, and on the same symmetry axis, is a drive piston. This is 3 inches in diameter and held stationary by shear pins. These pins shear when the driving pressure reaches 300 psig. The drive piston accelerates across the gap and strikes the protruding steel piston. This piston transmits the energy to the Sylgard, which behaves like a liquid having an air-filled void. The void collapses under the resulting pressure, and a hot spot forms against the explosive. Two sizes of bubbles are currently used, 0.030 inch and 0.060 inch radius hemispheres. The principal data acquired from this test are free run (gap) distances required for 50% probability of initiation. Pressure records contribute to the GO/NO-GO decision for each test.

The EARK-25 formulation (EAK with 25% RDX) was tested in the activator for comparison with previously obtained data for Comp B and TNT. Preliminary indications, based on a limited number of firings, are that EARK-25, Comp B, and TNT exhibit a similar level of sensitiveness, where "sensitiveness" is interpreted as a measure of the severity of conditions necessary to produce ignition.

FRATRICIDE (ROUND-TO-ROUND COMMUNICATION) TESTS

It is important to the Army to be able to ship artillery rounds in their standard pallet configuration with the confidence that, if one round inadvertently detonates, adjacent rounds will not, so that the detonation will not propagate throughout the shipment.

EARK-25 was tested in 105-mm shells. Figure 13 shows the test configuration.

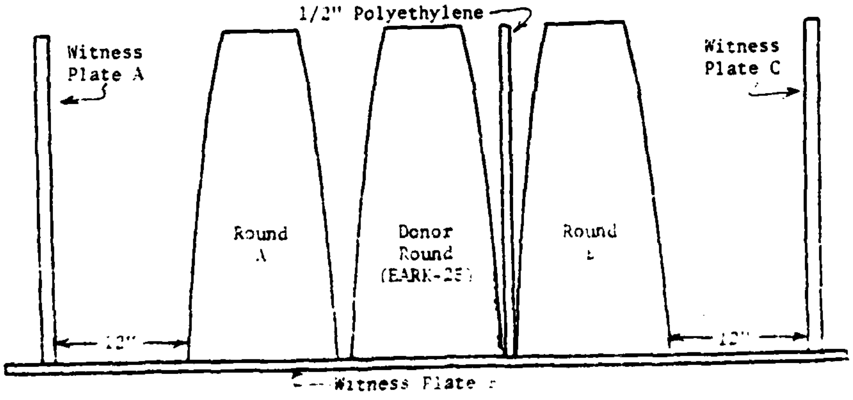


Figure 13. Configuration for EARK-25 Fratricide Test in 105-mm Shells.

A donor round of EARK-25 was placed between two acceptor rounds with the same configuration. Normal pallet spacing of ∿ 1/2 inch was preserved, with no cushion material on one side of the donor and 1/2 inch thick piece of polyethylene on the other side. This arrangement of three rounds was on a witness plate; two other witness plates were placed 12 inches away from each acceptor round, centered on the symmetry plane.

Based upon the results of this test, conclusions are that EARK-25 rounds will detonate if a neighboring round is detonated but will only react violently if protected with a 1/2 inch piece of polyethylene. These conclusions were based upon the damage done to the witness plates. Round A can be said to have detonated because:

a. Many small fragments struck witness plate A.

b. The dent formed by this round in witness plate B was almost as deep as that from the donor round.

c. Jetting action occurred at the junction between round A and the donor, caused by fragments striking each other (coming from opposite directions) and scattering onto the witness plate.

Round B can be said to have gone into a violent reaction, but not detonation, because:

a. Few high energy fragments struck witness plate C in contrast to those from round A onto witness plate A.

b. The dent formed on witness plate B was less than half the depth of the dents formed by the donor or round A.

c. Large fragments were recovered, which are not common in detonating rounds.

Two other fratricide tests were performed, one similar to that just described, but using CAB-lined 105-mm rounds and DARK-25 explosive. The ingredient ratios were the same as for EARK-25, but diethylene triamine was substituted for ethylene diamine. These rounds were considerably more sensitive than those filled with EARK-25.

The other fratricide tests were performed with 155-mm rounds and neat EAK explosive. These tests indicated that neat EAK has about the same sensitiveness to impact as TNT. This is more sensitive than is desired, so attempts are being made to plasticize EAK, as discussed earlier.

FRAGMENT SPEED TESTS

This test provides a measure of performance for an explosive. 105-mm shells were used, with EARK-25. Fragments were apertured by a heavy steel barricade; the windowed solid angle for fragment propagation terminated in celotex bales, for fragment capture. Precisely timed flash x-rays were used to generate multiple images of each fragment in the field of view. Fragment speed was calculated from image separation and flash interval. Fragment speed was determined as a function of launch angle measured from the projectile nose. The results are plotted in Figure 14 along with data from TNT and Comp B firings.

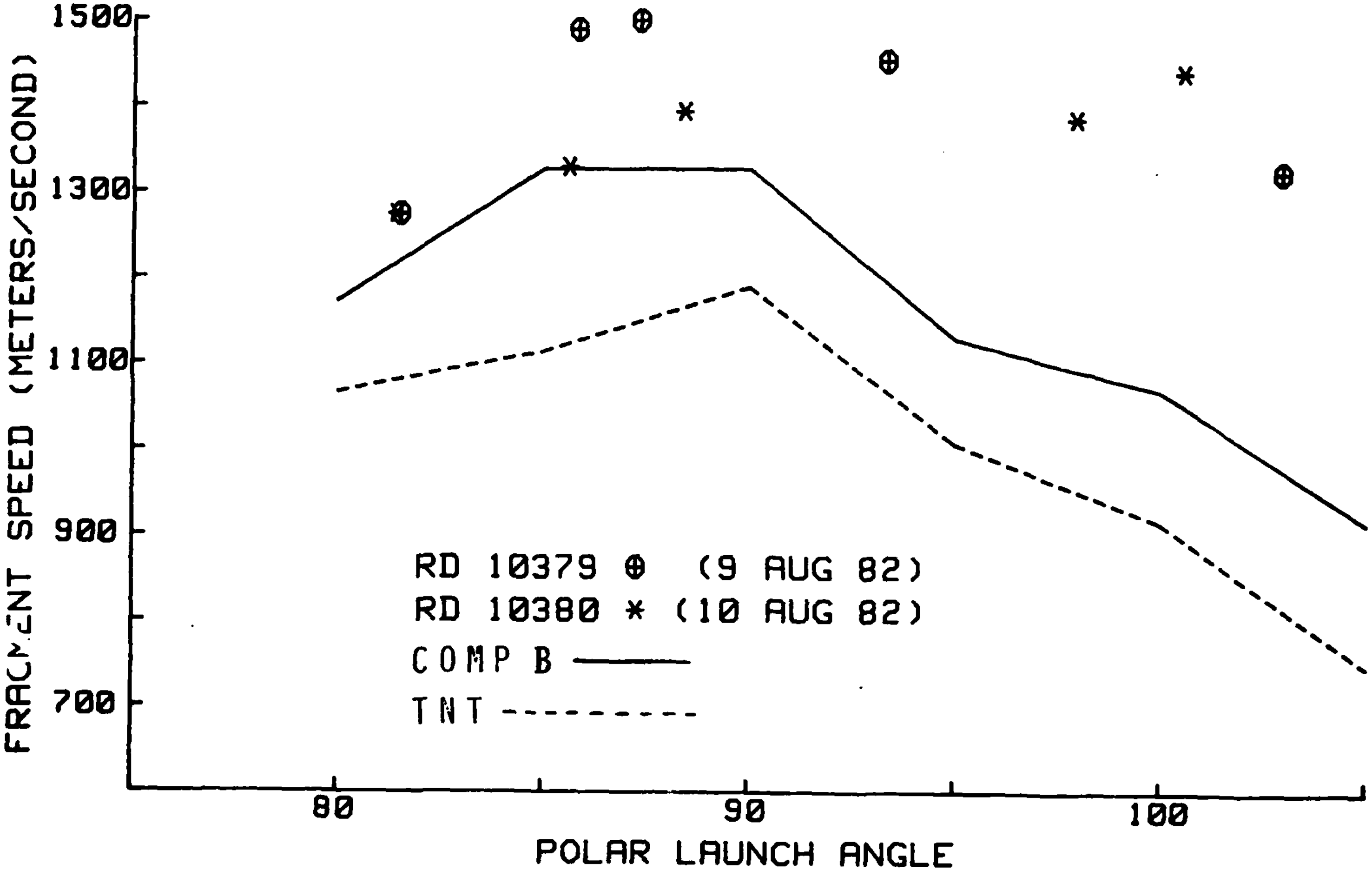


Figure 14. Fragment Speed From EARK-25 Filled 105 mm Rounds, Compared With That of Comp B and TNT.

Two shots were fired, and fragment histograms were plotted. Figure 15 represents these data. Many more small fragments were probably formed than were recovered, because of the poor recovery technique. Just under 50%, by number, of recovered fragments had masses of less than 1 gram, thus indicating a very brisant detonation. This was corroborated by the fragment x-ray data which indicated high fragment speeds. The graph of fragment speed versus polar launch angle showed a broad peak centered around 90°, similar in shape to the related plots for TNT and Comp B filled 105-mm rounds. Significantly, the fragment speeds for the EARK-25 filled rounds were 13% greater than for the Comp B filled rounds.

MISCELLANEOUS TESTS

A single, 1 inch diameter cylinder test was run, as were drop weight impact and friction tests. The cylinder test showed similar performance to composition-B, even for this small size; a more favorable comparison is expected for larger diameter cylinder tests. Friction and drop weight impact tests were run at Picatinny Arsenal. The 50% height for EARK-25, using the Type 12 Drop Weight Impact Tester, was 37 cm as compared to 45 for composition-B. EARK-25 showed no response to the friction pendulum test.

V. CONCLUSIONS

As stated earlier, the purpose of this work was to develop a new explosive based on eutectic blends of ammonium nitrate and ethylenediamine dinitrate and its homologues. As accomplishment of this purpose was approached, it became desirable to refine this purpose, to make it more specific, viz., to develop replacement explosives for TNT and Comp B. That for TNT could be EAK plus a sensitizer, and that for Comp B, EAK plus an enriching agent. This paper has described the learning process experienced by this laboratory in its attempt to do that, i.e., to develop intermolecular explosives as substitutes for TNT and Composition-B.

Of all the processes tried, the double direct seems most feasible. Remanent water and extremely low booster sensitivity are problems, both of which appear surmountable. An additional problem, associated with IMX materials produced by all processes, is that of incorporating, wetting, and suspending RDX long enough for the blend to solidify with a uniform distribution. Creaming as done by the Air Force[11] and as done by the Army, both appear to provide the capability of suspending particulate matter in EAK. Wetting agents have been found which aid the incorporation of RDX into EAK and which reduce the foaming experienced during water removal by vacuum stripping. RDX can be added to the EAK in its wet, as-shipped, condition, thus making the process safer. Work continues on finding the optimum processing aid for wetting, dispersing, and de-foaming.

Performance tests done here and elsewhere indicate that neat EAK, with an appropriate sensitizer, will be able to replace TNT, and that 75% EAK/25% RDX (EARK-25) will have performance superior to that of Comp B. Performance of EARK-25, particularly in terms of fragment speed produced in 105-mm rounds, is especially good. In fact, the performance is good enough to allow much latitude in sensitiveness reduction techniques without excessive concern about concomitant performance reduction. Its sensitiveness to impact with 25% RDX is greater than desired, being roughly equivalent to that of Comp B and

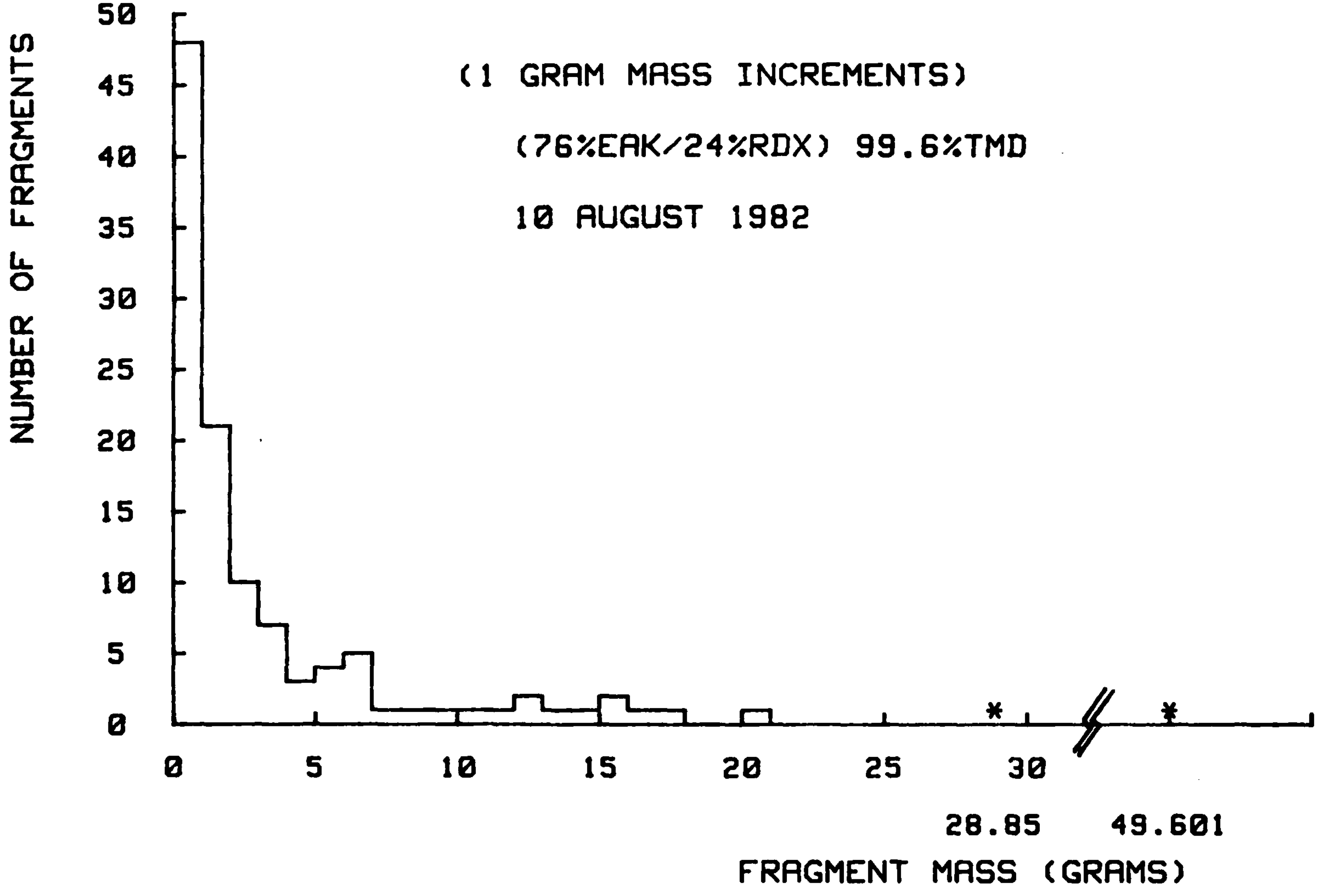


Figure 15. Fragment Histogram From an EARK-24 Filled 105 mm Round.

in some instances greater. To reduce this sensitiveness, the amount of RDX in the EARK will be reduced, and means of plasticizing the EAK will be pursued.

Although numerous factors are involved in the feasibility of any explosive fill in artillery shells, among the most important ones to evaluate in the early stages are performance, safety, and general producibility. To replace or augment existing fills, feasibility also requires some advantage over the extant ones. In these senses, it is considered that the evaluation indicates feasibility of the EA system, at least in shells of 105-mm or larger, and that the advantages which could accrue are significant.

ACKNOWLEDGEMENTS

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APPENDIX A

PROCEDURE FOR MAKING 30-KG MIX OF EAK BY THE DOUBLE DIRECT METHOD

APPENDIX A

PROCEDURE FOR MAKING 30-KG MIX OF EAK BY THE DOUBLE DIRECT METHOD

This procedure was performed at the BRL Hot Melt Facility on 23 Sep 81. Times are indicated on the left, followed by the associated comments. The experimental configuration was that of Figure 4.

Procedure

0905 Running tap water through kettle jacket (rate ∿ 1/2 liter/sec). Began adding 70% HNO_3 to kettle.

0921 Finished adding HNO_3

0923 Began stirring @ 20 psig

0926 Began adding AN. T = 20°C (Thermal well immersed). Poured pre-weighed bags of AN into transfer pail and dipped from that with a 1-liter beaker and poured into top port of 10-gal kettle.

0933 T = 12°C

0935 Finished AN addition. Began KN addition

Quantities:

AN	12.750 kg
KN	2.250 kg
HNO_3	10.26 liter
ED	5.44 liter

0937 T = 10°C

0943 Finished KN addition. T = 12°C

0953 Began ED addition

0958 T = 21°C

1004 T = 32°C. Cooling water still running.

1005 First liter ED added (1 liter/12 min → 83 ml/min)

1009 Began second liter ED addition. T = 32°C

1010 T = 33°C

1011 T = 34°C. Stirring still going well.

1013 T = 36°C

1017 T = 36°C. Appears to be holding.

1022 Completed second liter ED addition

1026 Began third liter ED addition. T = 31°C. Stirring @ 20 psig.

1033 T = 42°C. Finished third liter ED addition (7 min/liter → 143 ml/min)

1036 Began fourth liter ED addition.

1037 T = 41°C

1043 T = 44°C

1044 T = 45°C

1054 T = 49°C

1057 T = 50°C

1104 Finished fourth liter ED addition (1 liter/28 min → 35.7 ml/min), T = 51°C

1107 Began fifth liter ED addition. (Put 1.044 liter into seperatory funnel - need 396 ml more.) T = 50°C

1113 T = 52°C. ED flowing faster than for last addition.

1121 T = 54°C

1142 T = 59°C. Finished fifth liter ED addition, plus added 44 ml.

1144 Began last ED addition - 396 ml. T = 58°C

1147 T = 62°C

1151 Finished ED and all other additions. T = 62°C. Cooling water turned off.

1208 Steam started.

1213 88.5°C. Vacuum pump started - control valve partly closed.

1215 89°C. 14" Hg vac. Steam rate increased.

1218 100°C. 15" vac.

1223 101°C. 20" vac. Visible vapor and droplets in condensate trap.

1245 101°C. 21" vac. Water droplet rate increased in condensate trap.

1255 100°C. 22.5" vac. Vacuum increased - water rate increased. Turned vacuum back to 21".

1323 Bringing temp up to 105°C.

1325 T = 103°C. 21" vac. Very high vapor rate and surface of melt is turbulent (boiling).

1330 T = 105°C. 22" vac.

1347 T = 107°C. 21-1/2" vac. Melt still boiling.

1355 Bleeder valve on pump set at 1/8 turn open.

1359 T = 109°C. 21-1/2" vac. ∿ 1 drop/2 sec into condensate trap.

1420 Checked vacuum pump: fluid level at top of sight glass and fluid looks milky. Closed vacuum control valve at filter/trap. Drained pump oil. Connected aspirator. Tried to use it - had reverse flow. Kettle maintained ∿ 22" vac.

1435 Surface of melt is quiescent. Poured. Dime-sized patch of "solid" brown residue found in bottom of kettle.

Density after solidification:

$$\rho = 1.529 \text{ g/cm}^3$$

This is low density ∿ 92% TMD (1.665).

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